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RESEARCH REPORT



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FINAL REPORT

on

THE PROPERTIES OF POTENTIAL LOW-COST
HEAT-TRANSFER FLUIDS

to

SANDIA CORPORATION
LIVERMORE LABORATORIES

March 15, 1974

by

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The following abbreviations have been selected for use in this report. The English system of units which has gained recognition in engineering was adapted.

specific heat	Btu/(lb)(F)
coefficient of heat transfer	Btu/(hr)(ft) ² (F)
thermal conductivity	Btu/(ft) ² (hr)($\frac{F}{in}$)
heat transfer	Btu/hr
heat quantity	Btu
temperature	F
weight	lb
distance in flow	ft or in.
time	sec or hr
coefficient of volumetric expansion	F ⁻¹
density	lb/ft ³
volume	ft ³ /or gallon
viscosity	centistokes or seconds Saybolt Universal
vapor pressure	mm-Hg

SUMMARY, CONCLUSIONS AND RECOMMENDATIONS

A literature investigation has been carried-out wherein certain critical properties of several candidate heat-transfer fluids have been determined. The prime candidate fluids are Numbers 2, 4 and 5 fuel oil. In addition, two natural products, peanut oil and fish oil, have been evaluated. The critical properties sought include

- Cost
- specific gravity
- thermal expansion coefficient
- pour point
- specific heat
- thermal conductivity
- flash point
- viscosity
- vapor pressure
- thermogravimetric analysis (up to 600 F)
- additive action
- materials compatibility.

Cost data have also been accumulated for some commercial heat-transfer fluids.

The major findings of this investigation are the following:

1. The properties of the fuel oils vary widely with refiner and geographic location. There is a dearth of standards and those that exist are not necessarily adhered to.
2. Thermogravimetric analysis and sedimentation data for temperatures > 350 F are not available for any of the materials.
3. The fuel oils are probably not sufficiently stable for prolonged use at elevated temperatures.
4. The natural oils, peanut and fish oil, are promising candidates although their high-temperature properties are uncertain.
5. The fused salts are promising, but the corrosive effects are uncertain.

6. Commercial heat-transfer fluids are expensive relative to the fuel oils, but the greater stability and, hence longer life, may off-set the initial higher cost for synthetic fluids.
7. Jet fuels and hydraulic fluids may have the requisite stabilities at a favorable cost.

In order to fill in the gaps in the data and provide the basis for a critical design trade-off, the following tasks are recommended:

1. Differential thermal analyses at elevated temperatures must be carried-out for the fuel oils and the natural oils.
2. Closed- and open-system sedimentation rates and viscosity-change rates should be evaluated over long periods of time at elevated temperatures.
3. A life vs initial cost trade-off should be carried-out for the commercial heat-transfer fluids vis-a-vis the fuel oils and natural oils.
4. The suitability of eutectic salts, jet fuel and hydraulic fluids should be determined relative to system requirements.

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PURPOSE

The purpose of this study is to identify, collect and critically analyze the technical data pertinent to the thermophysical properties of fuel oils as heat-transfer fluids. The principal objects of the study were Numbers 2, 4, and 5 fuel oil. Otherwise, other candidate fluids were studied cursorily. These included peanut oil, fish oil, synthetic fluids, eutectic salts, hydraulic oils, and jet fuel.

RESEARCH METHOD

The study conducted in four phases:

- Information collection
- organization and review of the information
- critical analysis of the technical data
- recommendations for future studies.

The information collected through a review of the open literature, computer searches of several data banks (AEC, DDC, NTIS, CIRCOL), a scan of two abstract services and discussions with technical representatives of large refineries. The information survey yielded approximately 500 references from more than 100 different sources. Once collected, each piece of literature, or bit of technical data received from personal contact, or from the open literature, was critically analyzed relative to its contents.

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INTRODUCTION

In indirect heating units, heat from the combustion of fuel, electric energy, nuclear energy, solar energy or other sources, is transferred to a liquid, solid, or gas and carried by this material to the process or area where it is desired. Indirect heating systems, which date back to ancient times, make it possible to locate the heat source unit in the most convenient place, and supply the heat from that source, to a number of processes or areas where it is needed. Indirect heating systems also have an added advantage of close control of temperatures.

For the intended application, a candidate heat transfer agent must meet the following basic requirements:

1. Resist physico-chemical deterioration at the operating temperature over a long service life, to assure a clean efficient system
2. Retain good heat-transfer characteristics
3. Have a low vapor pressure at operating temperatures
4. Have a low viscosity to give low pumping losses and power requirements for circulation
5. Present no corrosion problems in the system
6. Be moderate in cost (taking into consideration initial cost and effective life).

The following properties were identified as critical in meeting these requirements:

1. Cost
2. Specific gravity
3. Thermal expansion

4. Pour point
5. Specific heat
6. Thermal conductivity
7. Flash point
8. Viscosity
9. Vapor pressure
10. Thermogravimetric analysis (TGA)
11. Additive action
12. Compatibility with other materials.

GENERAL DISCUSSION

The specific properties of Numbers 2, 4, and 5 fuel oil, and a vegetable oil (peanut oil) were identified and critically analyzed with respect to their utilization as heat-transfer fluids. The numbers of fluids investigated was limited by the scope of the project.

Properties identified and analyzed as being critical in the evaluation of the above materials as heat-transfer fluids included, but were limited to, cost, specific gravity, thermal expansion, pour point, specific heat, thermal conductivity, flash point, viscosity, and vapor pressure. Attempts were made to secure and evaluate data related to thermogravimetric analysis (to temperatures of 60 F), characteristic rates of bottom sediment formation (at 550 F in a closed system), and the use of additives to inhibit corrosion, sludge formation and bottom sediment.

In addition, special heat-transfer materials were reviewed and several materials were identified as candidate fluids for use in a closed system operating at temperatures between 500 to 600 F. The chemical composition and approximate cost of these heat-transfer fluids is provided. The possible use of jet fuel and hydraulic fluid is also discussed.

FUEL OILS

Introduction

Chemistry

Any petroleum product, whether it is crude oil, gasoline, lubricating oil, or fuel oil is essentially composed of hydrocarbons. There are many types of hydrocarbons found in petroleum products (fuel oil) but most of them fall into four main classes; paraffin, aromatic, naphthenic, and olefinic. Sulfur is also present in varying amounts in all crude oils and in fuel oils in varying amounts. The presence of sulfur, even in small quantities - 1 % or less, can be quite troublesome to the refiner, distributor and consumer.

The following is a brief review of the different properties the aforementioned hydrocarbons show.

Paraffinnic Hydrocarbons. have a much higher specific gravity or lower API gravity than naphthenic, olefinic or paraffin hydrocarbons. They are very stable to heat but are chemically active to a limited degree. This class of hydrocarbon is present in cracked oils in a greater percentage than in straight run oils, which is the reason cracked oils are heavier.

Naphthenic Hydrocarbons. are extremely stable compounds, and in most cases are more stable than the paraffins.

Olefinic Hydrocarbons. are more active chemically than the other three classes of hydrocarbons. They are not found in large amounts in straight run distillates but are present in larger quantities in cracked oils. Olefins under subject to oxidation or polymerization form gums. Formerly, all oils were straight run but today modern refining methods produce mostly cracked oils.

Cracked oils can be produced by two methods, thermal cracking and catalytic cracking. The latter method has proven to be most efficient and is the more modern one. Catalytic cracked fuel oils are superior in some ways to thermally cracked and straight run distillates. They are more stable to chemical change by oxidation because of the aromatic compounds being present in greater percentage than in straight run or thermal cracked fuels. Catalytic cracked fuels are also more stable at elevated temperatures than fuels produced by the straight run or thermal cracking process.

Standardization

The National Bureau of Standards and The American Society for Testing Materials have standardized five grades of fuel oils. They designated these grades as Nos. 1, 2, 4, 5, and 6. In 1948, No. 3 fuel oil was deleted from the classes. Although there are only five basic grades of fuel oil today, many organizations have established variations in the specifications. For example, the U. S. Navy has sets of specifications fuel oils must meet before they can be purchased and used for various naval applications.⁽¹⁾ A description of the five fuel oil classes (Nos.) and the National Bureau of Standards specifications for the fuel oils is presented in Table 1.

It is difficult to judge the quality of fuel oil. A specific fuel might be quite acceptable in one geographic location of the United States and be unacceptable for use in a different area. Specifications of specific fuel oils provide for variations in the product. Figure 1 identifies the geographical areas established as refining districts by the United States Bureau of Mines. Fuel oil purchased from a supplier within a refining district will probably be more uniform in composition because of special blending to meet regional conditions. Surveys taken in the various marketing areas have identified fuels which do not meet even the loose specifications established by the National Bureau of Standards for Fuel Oils. The results of the chemical analysis of sulfur content in various grades of fuel oil is shown in Table 2.

(1) Schmidt, P. F., Fuel Oil Manual, The Industrial Press, New York, 1958, pp 159-161.

Table 1. National Bureau of Standards Specifications for Fuel Oils (1)

Number	Grade of fuel oil ^b Description	Flash point, °F	Pour point, °F	Water and sediment, %	Carbon residue on 10% residuum, %	Ash, %	Distillation temperatures, °F			Viscosity				Gravity, °API				
							10% point	90% point	End point	Saybolt		Kinematic centistokes at—						
										Universal at 100° F	Furol at 122° F	100° F	122° F					
1.....	Distillate oil intended for vaporizing pot-type burners and other burners requiring this grade. ^a	min 100 or legal	max 0	max Trace	max 0.15	max	max 420	max	max 625	max	min	max	min	max 2.2	min 1.4	max	min	min 35
2.....	Distillate oil for general purpose domestic heating for use in burners not requiring No. 1.	100 or legal	4 20	0.10	0.35		(*)	675		40				(4.3)				36
4.....	Oil for burner installations not equipped with preheating facilities.	130 or legal	20	.50		0.10				125	45			(28.4)	(5.8)			
5.....	Residual-type oil for burner installations equipped with preheating facilities.	130 or legal		1.00		.10					150	40		(32.1)	(81)			
6.....	Oil for use in burners equipped with preheaters permitting a high-viscosity fuel.	150 or legal		2.00								300	45		(638)	(92)		

^a Recognizing the necessity for low-sulfur fuel oils used in connection with heat treatment, nonferrous metal, glass and ceramic furnaces, and other special uses, a sulfur requirement may be specified in accordance with the following table:

Grade of fuel oil	Sulfur, max, percent
No. 1.....	0.5
No. 2.....	1.0
Nos. 4, 5, and 6.....	No limit

Other sulfur limits may be specified only by mutual agreement between the buyer and seller.

^b It is the intent of these classifications that failure to meet any requirement of a given

grade does not automatically place an oil in the next lower grade unless in fact it meets all requirements of the lower grade.

^c No. 1 oil shall be tested for corrosion in accordance with par. 15 for 3 hours at 122° F. The exposed copper strip shall show no gray or black deposit.

^d Lower or higher pour points may be specified whenever required by conditions of storage or use. However, these specifications shall not require a pour point lower than 0° F under any conditions.

^e The 10-percent point may be specified at 440° F. Maximum for use in other than atomizing burners.

^f The amount of water by distillation plus the sediment by extraction shall not exceed 2.00 percent. The amount of sediment by extraction shall not exceed 0.50 percent. A deduction in quantity shall be made for all water and sediment in excess of 1.0 percent.

(1) Guthrie, V. B., Petroleum Products Handbook, McGraw-Hill Book Company, Inc., 1960, p 18.

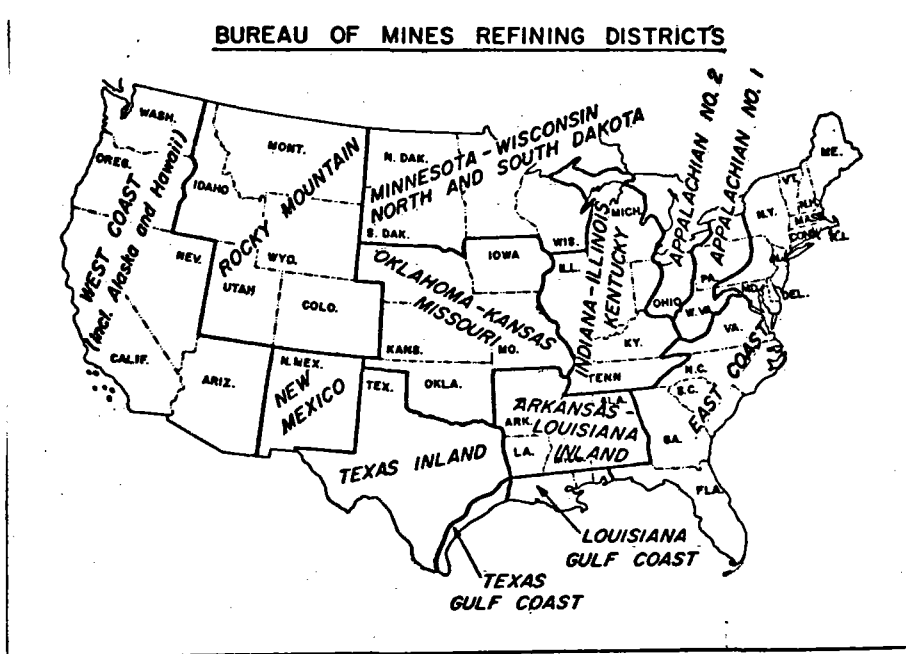


FIGURE 1. MAP OF BUREAU OF MINES REFINING DISTRICTS⁽¹⁾

(1) Bureau of Mines Yearbook, Crude Petroleum and Petroleum Products, United States Department of Interior, U. S. Government Printing Office, Washington, D. C., 1971, p 5.

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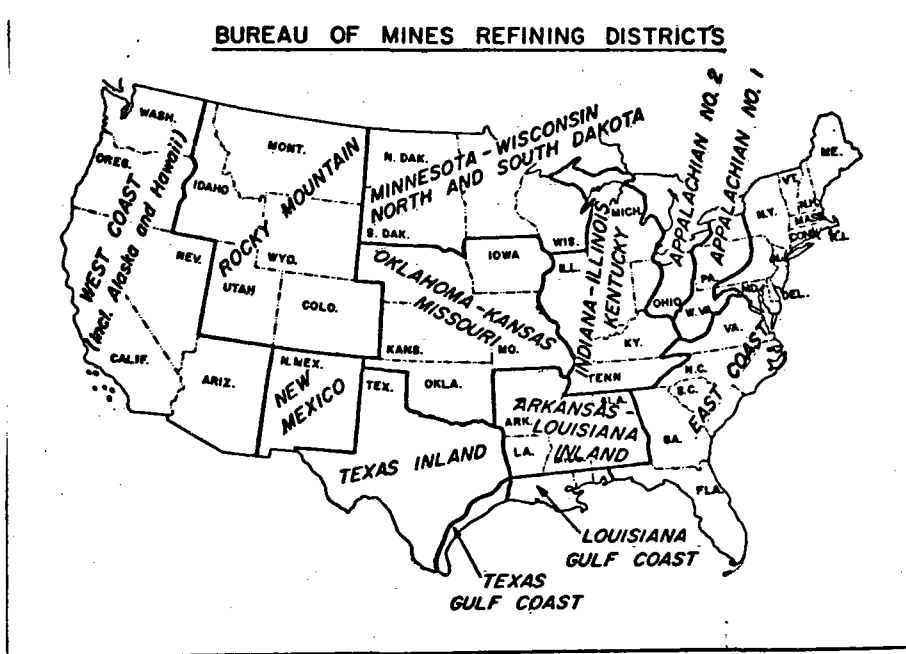


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(1) Bureau of Mines Yearbook, Crude Petroleum and Petroleum Products, United States Department of Interior, U. S. Government Printing Office, Washington, D. C., 1971, p 5.

TABLE 2. SULPHUR CONTENT OF TYPICAL SAMPLES OF
VARIOUS GRADES OF FUEL OIL

Grade	Sulphur Content, percent
No. 2	0.16 to 0.42
No. 4	0.35 to 0.75
No. 5	0.69 to 1.02

Specific Properties

Costs

Wholesale prices of fuel oils had increased well in advance of the Mid-East crisis. In 1967, electric utilities began switching to fuel oil for the generation of electric power. In three years (1969-1972) direct use of distillate fuels by electric utilities had tripled.⁽¹⁾ Fuel oil producers cannot produce the volumes of fuel oil needed by the consumer today. Due to the demand for fuel oil, the drastic increases in crude oil costs, and the continued raise in refining and distribution costs, the availability and cost of this product in the future are questionable at this time.

An excellent reference to acquire daily quotations on fuel oil prices is "Platt's Oilgram Price Service" published by McGraw-Hill Publishing Company. In this reference, fuel prices of refiners and terminal operators (FOB refiners and terminals for fuel oil No. 2) ranged from 16.5¢ to 37.7¢/gallon on December 14, 1973. Fuel oil No. 4 was quoted at 19.91¢ to 23.3¢ per gallon and prices in Chicago for fuel oil No. 5 with a maximum of 1 percent sulfur content ranged from 18.5¢ to 23.5¢ per gallon during the same day.

(1) Lynch, R. D., "What Caused Heating Oil Shortage", Fuel Oil and Oil Heat, February 1974, Vol. 33, Nr. 2, pp 38-40.

Specific Gravity and Density

The density of a material is its weight per unit volume at a given temperature. Specific gravity is defined as the ratio of the density of a given substance to the density of a reference substance. Specific gravity is a dimensionless number universal to all systems of units. In the English system, for liquids, water at 60 F and 14.7 psi is used as the reference material.

There are numerous references to the specific gravity of fuel oils in the literature. Table 3 presents the range of specific gravity of fuel oils Nos. 2, 4, and 5 based the rating in °API. A graph showing specific gravity and pounds per gallon vs. °API gravity of petroleum distillates is included in Appendix A of the report.

TABLE 3. SPECIFIC GRAVITY OF FUEL OILS

	Gravity, °API	Specific Gravity, 60F/60F
Fuel Oil No. 2	30.2 - 45.3	.8757 - .8000
Fuel Oil No. 4	16.9 - 29.4	.9581 - .8790
Fuel Oil No. 5	10.5 - 19.7	.9959 - .9353

Thermal Expansion

Like all fluids, fuel oils expand upon heating. The extent of expansion is dependent upon the volume of fluid involved, the number of degrees heated, and the particular fuel oil. The expansion is considerable, and means must be provided in the system for it. For example, if 100 gallons of No. 5 fuel oil were heated from 50 F to 450 F it would become approximately 114 gallons.

The thermal expansion coefficient based upon °API rating of fuels marked in the United States during 1973 is given in Table 4. Figures are included in Appendix B present data relating the thermal expansion of liquid petroleum fractions by °API at 0, 250, 500, 1000, and 1500 lbs/in² G. to temperature. The graphs cover a temperature range of 0 F to 1000 F.

TABLE 4. THERMAL EXPANSION COEFFICIENT FOR CANDIDATE HEAT-TRANSFER FLUIDS

	Range °API, 1973	Coefficient of Thermal Expansion
Fuel Oil No. 2	30.2 - 45.3	.00040 - .00050
Fuel Oil No. 4	16.9 - 29.4	.00040
Fuel Oil No. 5	10.5 - 19.7	.00035 - .00040

Pour Point

The National Bureau of Standards Specifications for Fuel Oils lists the pour point for No. 2 fuel oil to be 20 F; however, variations are permitted whenever specified. A 20 F pour point is also listed for fuel oil No. 4 in the standards. No pour point was specified for No. 5 fuel oil in the National Bureau of Standard Specifications. Table 5 gives the pour point which meets ASTM D396 maximum requirements⁽⁵⁾.

TABLE 5. POUR POINT OF FUEL OILS NOS. 2, 4, AND 5

	Maximum Pour Point, ⁽¹⁾ F
Fuel Oil No. 2	20
Fuel Oil No. 4	20
Fuel Oil No. 5	70*

*Highest pour point No. 5 fuel reported, no maximum temperature given in requirements.

(1) Schmidt, P. F., Fuel Oil Manual, The Industrial Press, New York, 1958, p 18.

Flash Point and Vapor Pressure

The flash point of fuel oils sold in the United States varied with the grade (No.) distillation range, geographic region of the sale, and refiner. In 1973, flash points of No. 2 fuel oils marketed in the United States were reported to have ranged from 126 F to 204 F. The flash point of No. 4 fuel oils sold in the United States during 1973 was reported to have run from 142 F to 240 F. Refineries during the same year reported the sale of two grades of No. 5 fuel oils - a light grade whose flash point ranged from 156 F to 336 F, and a heavy grade whose flash point ran quite higher.

There is a relationship between vapor pressure and flash point. Based upon the flash point of fuel oils sold in the United States during 1973, and utilizing the relationship, the following ranges of vapor pressure were estimated for distillate Nos. 2, 4, and 5 fuels. The vapor pressure of No. 2 fuel was approximately 5mm of Hg at 100 F; No. 4 ranged from 2.5 to 3.5mm of Hg at 100 F; and the range of No. 5 distillate fuel was approximately 0.1 to 2.4mm of Hg at 100 F.⁽¹⁾

Table 6 provides a review of the ranges of flash point and vapor pressures of fuel oils Nos. 2, 4, and 5 based upon the National Bureau of Standards' report of fuel oils sold in the United States during 1973. It must be emphasized that these data will not hold true for fuel oil sales today but can only serve to indicate ranges of their physical properties.

TABLE 6. FLASH POINT AND VAPOR PRESSURE OF
CANDIDATE HEAT-TRANSFER FLUIDS

	Flash Point F	Vapor Pressure at 100 F, mm-Hg
Fuel Oil No. 2	126 - 204	5
Fuel Oil No. 4	142 - 240	2.5 - 3.5
Fuel Oil No. 5	156 - 336	0.1 - 2.4

(1) Aremo, Gulf Oil Research and Development Center, Harmarville, 1972, Discussions.

Specific Heat

Figure 2 shows the specific heat for hydrocarbon liquids in Btu/lb/F by °API rating for temperatures ranging from 0 F to 1000 F.

Thermal Conductivity

An important property to be considered in the valuation of a heat-transfer agent is the material's thermal conductivity. The thermal conductivity data for fuel oils Nos. 2, 4, and 5 given in Table 7 were calculated using the following equation:

$$K = \frac{0.813}{d} [1 - 0.0003 (t - 32)]$$

in which K = thermal conductivity in Btu per hour, ft² and degree F/in.
d = specific gravity of the liquid at 60/60 F and t = temperature in degrees F.
The equation is based on experimental results obtained at atmospheric pressure.

TABLE 7. THERMAL CONDUCTIVITY OF CANDIDATE HEAT-TRANSFER FLUIDS

	Range Specific Gravity, 60/60 F	Range, t = 100 F Btu/hr/ft ² (F)/in	Range, t = 500 F Btu/hr/ft ² (F)/in
Fuel Oil No. 2	.8000 - .8757	0.91 - 1.0	0.80 - 0.87
Fuel Oil No. 4	.8790 - .9581	0.83 - 0.91	0.73 - 0.80
Fuel Oil No. 5	.9353 - .9959	0.80 - 0.85	0.70 - 0.75
Peanut Oil	.917 - .926	0.86 - 0.87	0.75 - 0.76

It is interesting to compare the results obtained for peanut oil with those for fuels, but there are no experimental results to verify the data.

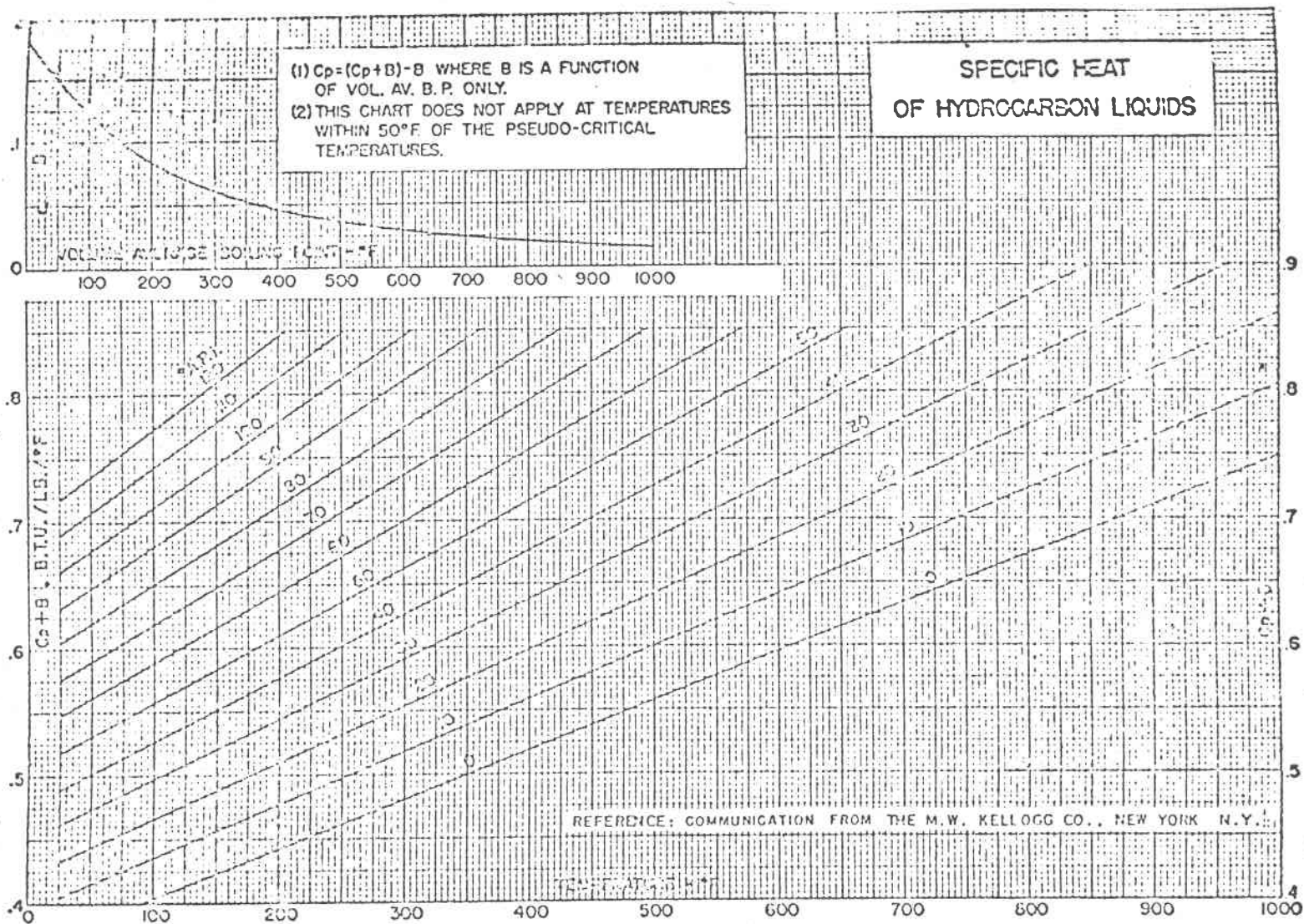


FIGURE 2. SPECIFIC HEAT OF HYDROCARBON LIQUIDS ⁽¹⁾

(1) From Maxwell, J. B., "Data Handbook of Hydrocarbons", D. Van Nostrand Company, Inc., New York (1950), p 93.

Viscosity

The viscosity of fuel oil is the measure of its resistance to flow. Upon heating the viscosity of fuel oils decreases. Table 8 shows the relation of viscosity to temperature. As the oil thins out upon heating, it is easier to pump.

TABLE 8. RELATION OF VISCOSITY AND TEMPERATURE OF FUEL OIL⁽¹⁾

Temperature, Deg. F.									
100	120	140	160	180	200	220	240	260	280
Viscosity, Seconds Saybolt Universal									
100	74	60	52	46	42	40	38	36	35
200	130	90	68	57	49	45	41	39	36
300	170	110	78	62	52	46	42	40	37
400	230	140	98	74	60	52	47	43	40
500	290	180	120	87	69	58	51	46	43
600	340	215	140	100	78	64	56	49	45
700	400	240	160	120	82	67	57	50	46
800	440	265	170	125	88	70	52	47	43
900	480	290	180	130	90	72	60	53	47
1000	540	300	190	140	92	76	64	55	48
1500	750	420	250	160	110	82	67	57	51
2000	1020	575	350	220	150	110	85	70	60
2500	1248	680	390	260	165	117	89	72	61
3000	1400	750	415	285	175	122	92	74	62
3500	1640	850	475	300	187	128	95	76	63
4000	1800	900	500	315	190	132	98	77	64
4500	2040	1020	565	330	208	142	104	80	66
5000	2200	1100	600	350	220	150	108	83	68
6000	2600	1250	675	380	240	160	115	88	71
7000	2900	1400	750	420	260	175	125	93	74
8000	3200	1550	820	460	280	185	138	97	77
9000	3600	1700	875	500	295	195	145	102	79
10000	4000	1900	950	555	312	210	150	106	82

Figure 3, "Variation of Fuel Oil Viscosity With Temperature" is a graph which shows the viscosity change with regard to change in temperature for Nos. 4 and 5 fuel oils. Table 9 identifies the viscosity range of fuel oils sold on the United States Market in 1973.

(1) Schmidt, P. F., Fuel Oil Manual, The Industrial Press, New York, 1958, p 43.

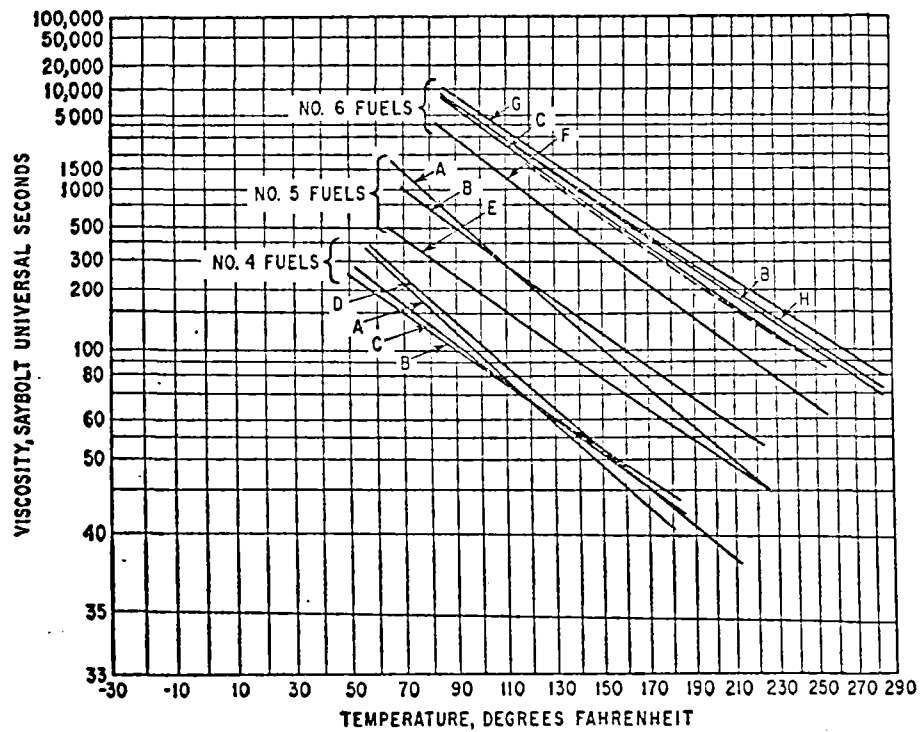


Figure 3. Variation of Fuel Oil Viscosity With Temperature⁽¹⁾

(1) From Guthrie, V. B., Petroleum Products Handbook, McGraw-Hill Book Co., New York (1960), pp 8-24.

TABLE 9. VISCOSITY OF CANDIDATE FUEL-OIL HEAT-TRANSFER FLUIDS

	Viscosity in Centistokes	
	100 F	450 F(*)
Fuel Oil No. 2	1.8 - 4.1	.4 - .51
Fuel Oil No. 4	6.3 - 28.7	.76 - 1.2
Fuel Oil No. 5 Light	17 - 57.8	1.1 - 1.4
Heavy	- 151	- 1.9

(*) Estimated

Thermogrammetric Analysis (TGA)

There is an abundance of technical literature related to volume-temperature relationships for petroleum products; however, no reliable TGA analysis information was uncovered by the literature search or provided by the refiners.

Rates of Bottom Sediment, Sludge Formation and Effect of Additives

No data were located that would allow an estimation of the characteristics and rates of bottom sediment and sludge formation of fuel oils at temperatures approaching 500 F. There was also a dearth of information related to sediment or sludge formation of distillate oils in a closed system. There is, however, considerable evidence of instability of fuel oils when exposed to the atmosphere at only slightly elevated temperatures. It is interesting to note that chemical analysis of fuel oil sludges show 12 percent oxygen content while analysis of the parent oil indicate only a trace of oxygen.

Organic-sediment is a product of oxidation and chemical reactions involving unsaturated hydrocarbons and reactive organic compounds of sulfur, nitrogen, and oxygen, which are found in small amounts in petroleum distillates.

Inorganic materials, such as rust, are often called "sediment" in addition to organic sediment. Refiners are asked to produce fuel oils having very low concentrations of organic sediment. However, the distribution system often allows contamination of the product by various sources.

A chemical reaction between copper (from either copper or brass metal that comes in contact with the fuel and mercaptans in the fuel), is thought to be associated with mercaptide gel formation. The copper mercaptide is believed to act as a gelling agent and forms the gel in contact with small amounts of water and oxygen. It appears that manufacturers can (and many do) produce a relatively clean product. However, in the process of storage and distribution to the user the fuel becomes contaminated. The National Bureau of Standards Specifications for Fuel Oils list maximum water and sediment content for Fuel Oil No. 2 to be 0.10 percent, Fuel Oil No. 4 to be 0.50 percent, and Fuel Oil No. 5 to be 1.00 percent. Home users of fuel oil report an increase in the number of plugged filters in early fall. The problem is caused by an increase of sediment and sludge which has collected during storage of the fuel over the summer months. Studies also indicate increases in sludge formation and bottom sediment when blends of fuels are used. Petroleum refiners blend or mix straight-run, catalytically cracked, and thermally cracked fuels and market these products throughout the United States.

In review, no information was detected that would identify rates of bottom sediment and sludge formation at elevated temperatures (400 F - 500 F) in a closed system. It was also determined by direct contact with refiners that the refiners or suppliers are not aware of the thermal stability of their fuel oils at elevated temperatures in closed or open systems.

Fuel oil additives are commonly used to help control the stability of distillate fuels. The composition of these additives varies widely and includes sulfonates, polymers, amines, metal phenolates, and combinations of these. Some are effective in a wide variety of fuels while others may be effective in only a few fuels. Detergent-dispersed additives do not prevent the formation of sediment or sludge but rather keeps the sediment well dispersed

in the oil and reduces particle size of the sludge. Sediment-inhibiting additives (oxidation inhibitors) actually eliminate or reduce the formation of organic sediment in fuels. In practice, many heating-oil additives are really bifunctional - providing both oxidation inhibition and a dispersant function.

A critical evaluation of fuel additive effectiveness is an extremely complex problem and an indepth analysis of the large volumes of literature and technical data pertaining to this problem is far beyond the scope of this project. A scan of the recent Chemical Abstracts did, however, produce several pertinent abstracts related to the problem and patents for new additives. The abstracts have been reproduced and are included in Appendix B of the report.

PEANUT OIL AND FISH OIL

Introduction

In view of the shortage of fuel oil, attempts were made to investigate ten additional heat-transfer fluid candidates - a vegetable oil (peanut oil) and an animal oil (fish oil).

The literature pertinent to the thermophysical properties was collected and reviewed in detail even though not in the scope of the project. The technical data related to peanut oil and fish oil were limited in volume and quality.

Peanut Oil

General Discussion. It appears that researchers have concentrated on developing new products from the peanut which can be placed in a box or wrapper and sold and have devoted little effort to the investigation of the specific properties of the peanut or its oil.

Investigators have been interested in peanut oil stability. It appears, however, from discussions with processors of the oil and a review of the literature, that investigations have been restricted to the evaluation of thermal stability for peanut oil under conditions found in the kitchen. The amount of unsaturated and saturated fatty acids varies with the varieties of nuts from which the oil was extracted. A correlation exists between the linoleic and oleic acid content and the stability of the oils. The Runner variety of peanuts contain less linoleic acid and have a correspondingly higher order of stability than do oils from either Spanish or Virginia peanuts. The stability of peanut oils can be increased by hydrogenation or by the addition of additives. No information was present that would identify rates of sludge formation, bottom sediment or thermal stability above 440 F; nor was information available to evaluate the thermal stability of peanut oil in a closed system at elevated temperatures. The information available pertinent to additives for peanut oil for the purpose of increasing thermal stability pertained to those which decrease oxidation in an atmospheric environment.

No flash point was identified for peanut oil. However, I. G. Woodroff in his book, Peanuts: Production, Processing, Products, reported that peanut oil exposed to the atmosphere would begin to smoke at approximately 440 F.

Physical Properties. The review of the literature and contact with technical people associated with the peanut industry identified limited technical data relevant to the chemical and physical properties of peanut oil.

The Dothan Oil Mill Company, Dothan, Alabama, and Gold Kist, Inc., of Atlanta, Georgia, gave identical quotes for peanut oil, \$3.19 per gallon. Mr. J. B. Roberts, president of the Dothan Oil Mill Company, suggested that the price of peanut oil is inflated at the present time. He expects drastic reductions in peanut oil prices once the present crop has been harvested. Exports of domestic supplies to foreign markets to supplement a bad harvest in South America is given as the reason for the present elevated price.

Peanut oils like fuel oils vary in compositions depending on source of the crude material from which it is refined. The specific gravity of peanut oils is reported to range from .910 to .915 at 60 F.

A 14 F pour point has been reported for peanut oils. Table 10 presents the specific heat for peanut oil at various temperatures.

TABLE 10. SPECIFIC HEAT AT VARIOUS TEMPERATURES FOR PEANUT OIL

Peanut Oil	
Temperature, F	Specific Heat
86	.495
177	.539
250	.565
342	.588
410	.590
427	.595
483	.633
520	.657

The thermal conductivity of peanut oil has been reported to be .004 calories per second per centimeter² per degree centigrade at 19 C, and its viscosity is reported to be 217 SSU at 100 F. No technical data were identified relative to the oils vapor pressure or TGA.

Chemical analysis of peanut oil tells us that its chemical composition is not uniform from batch to batch and that composition varies from variety of source nut, from field to field and from season to season. The composition of oils extracted from two varieties of nuts is presented in Table 11.⁽¹⁾

TABLE 11. COMPOSITION OF PEANUT OILS BY VARIETY

COMPOSITION OF PEANUT OIL ¹		
Glycerides	Varieties, %	
	Spanish	Virginia
Unsaturated		
Oleic $C_{18}H_{33}O_2$	52.9	60.0
Linoleic $C_{18}H_{31}O_2$	24.7	21.6
Saturated		
Palmitic $C_{16}H_{32}O_2$	8.2	6.3
Stearic $C_{18}H_{36}O_2$	6.2	4.9
Arachidic $C_{20}H_{40}O_2$	4.0	3.3
Lignoceric $C_{22}H_{44}O_2$	3.1	2.6
Unsaponifiable matter	<u>0.2</u>	<u>0.3</u>
Total	99.3	99.6

¹Calculated as simple triglycerides.

Table 12, identifies the usual range of characteristics for oils extracted from the Spanish and Runner varieties of peanuts.

(1) Woodroff, J. G., Peanuts: Production, Processing, Products, The Avi Publishing Company, Inc., Westport, Connecticut, 1973, p 256.

TABLE 12. CHARACTERISTICS OF PEANUT OIL

PEANUT OIL					
	Usual Range	AOCS	British Standards	Specimens	
				Spanish	N.C. Runner
References		9	27	96	96
Acid value	0.08-6		(a)	1.5	1.5
Saponification value	188-195	188-195	188 min.		
Iodine value	84-102	84-100	82-99	94.3	93.1
Thiocyanogen value	67-73	63		68.4	70.9
Hydroxyl value	2.5-9.5	8.6-9.6			
R-M value	0.2-1.0	Below 0.5			
Polenske value	0.2-0.7	Below 0.5			
Unsaponifiable (%)	0.2-0.8	Below 1	0.8 max.	0.64	0.71
Refractive index, n_D , 25°C	1.466-1.470	1.467-1.470	1.468-1.472 ^b	1.4683	1.4681
Refractive index, n_D , 40°C	1.4605-1.4645				
Specific gravity, 15/15°C		0.917-0.921	0.917-0.919 ^c		
Specific gravity, 25/25°C	0.910-0.915	0.910-0.915			
Titer (°C)	26-32	26-32			

^a To be agreed between purchaser and vendor. "The acidity of good commercial quality ground nut oil may range from hardly appreciable amounts up to 5%, calculated as oleic acid, according to season or district of origin."

^b Refractive index at 20°C.

^c Specific gravity at 15.5/15.5°C.

Conclusions. Peanut oil, like fuel oil, varies in chemical content and physical properties from batch to batch. The characteristics of the refined peanut oil will, however, remain relatively uniform for oil of a particular nut variety as compared to fuel oils. Technical data relative to the stability of peanut oil in the 500 F to 600 F range have not been identified nor has sludge formation or rates of sediment at elevated temperatures. Evaluation of the value of peanut oil as a candidate heat-transfer fluid cannot be made until gaps in the technical literature have been filled.

FISH OIL

Introduction

An animal oil (thermofluid), developed by Caliqua, S. A., Paris, is recommended for use as a heat-transfer fluid for operation in open systems at 400 F to 650 F. One possible disadvantage to the use of thermofluid as a heat-transfer fluid is the requirement to purchase supplies of the material from a foreign market.

Important Physical Properties

Thermofluid sells for under \$5.00 per gallon measured at 60 F and 16.696 psia. The density of thermofluid is 47.3 lb/ft³ at 200 F and this parameter decreases upon heating to 37.9 lb/ft³ at 600 F. No coefficient of thermal expansion was identified in the literature for thermofluid nor was information related to thermogravimetric analysis identified. The pour point is reported in the literature as 14 F.

The specific heat for thermofluid increases with an increase in temperature. At 100 F, the specific heat for thermofluid is 0.485 Btu/(lb)(F) and this increases to 0.8000 Btu/(lb)(F) at 600 F. The thermal conductivity of thermofluid is a linear function of temperature. At 212 F, the thermal conductivity is 0.0806 Btu/(hr)(ft)²(F)/in, and which increases to 0.0699 Btu/(hr)(ft)²(F)/in, at 662 F.

As with other fluids, the viscosity of thermofluid decreases with increased temperature. Its viscosity is reported as 15.5 centipoises at 150 F and decreases to 1.0 centipoises at 285 F. Thermofluid's vapor pressure does not rise above atmospheric pressure within the recommended operating range (400 F to 650 F).

Thermofluid remains stable at high temperatures and is used with most common structural materials. No special cautions appear in the literature with regard to handling techniques or danger of explosion.⁽¹⁾

(1) Geiringer, P. L., Handbook of Heat Transfer Media, Reinhold Publishing Corporation, 1962, pp 126-131.

Conclusions

Thermofluid appears, at first glance, to be an excellent candidate for a heat-transfer fluid to operate in closed systems at temperatures ranging from 500 F to 600 F. It must be pointed out, however, that the only real data which can determine the true operation properties in a system must come from direct observations of the fluids under simulated or real operating conditions.

OTHER HEAT-TRANSFER CANDIDATES

Recognized Heat-Transfer Fluids

Naphthalenes, alkyl naphthalenes, o-dichlorobenzene (Dowtherm E), diphenyl-diphenyl oxide (Dowtherm A), an isometric mixture of perfluoro amine, $(C_4F_9)_3N$, an isometric mixture of perfluoro cyclic ether, $C-C_8F_{16}O$ and p-methyl isopropyl benzene (para-cymene), are materials recommended for use as heat-transfer agents in the vapor state and thus were not investigated for this project. ⁽¹⁾ Several fluids covered function well in the 500-600 F temperature range. Some, however, do present special problems due to their corrosive nature. If Du Pont's Hitec were utilized as a heat-transfer fluid for example, special metals would have to be used in the fabrication of the systems to prevent corrosion. Hitec is, however, moderate in cost (\$16.20 per 100 pounds).

The Mobil Oil Corporation has several mineral-oil-based products for use as heat-transfer fluids that are reasonably priced. The products include Mobiltherm Light, Mobiltherm 600, Mobiltherm 603, and Mobiltherm 605. Mobil distributors report that Mobiltherm 600 will be removed from the market. The thermophysical properties of Mobiltherm 603 and 605 appear to be very similar at first glance and either material - depending upon availability of the supplier - should perform well as a fluid in the 450 F to 600 F range. The District Sales Office for Mobil Oil, located in Columbus, Ohio, reported the price of Mobiltherm 603 to be \$1.14/gallon in 50 gallon drums.

Humble Therm 500 (a mixture of aromatic hydrocarbons) is a moderately priced heat-transfer fluid which is also recommended for operation at temperatures up to 600 F in closed systems.

(1) Geiringer, P. L., Handbook of Heat Transfer Medium, Reinhold Publishing Corporation, New York, 1962, pp 67-101.

The Dow Corning Corporation of Midland, Michigan, produces several silicone fluids which are recommended for use as heat-transfer fluids serviceable at temperature ranges of -40 to 600 F in closed systems. These fluids, however, range in price from \$0.96 to \$7.27 per pound. Dow Corning 200 fluid is supplied in six different viscosities: 50, 100, 350, 500, 1000 centistokes at 77 F. The fluid is a dimethyl siloxane polymer which shows little change in physical properties over a wide temperature span. It is recommended for use at temperatures above 400 F, however, no information was identified that would recommend use in the 500 to 600 F range in a closed system. In fact, recognized authorities tell us additives must be used in this fluid before it can be used above 450 F.

Table 13, identifies several heat-transfer fluids which have proven serviceable in operating systems and classifies the fluid by composition. Prices of many chemical compounds change from day to day from source of supply and for geographic area. Because of the changing price structure in today's economy a price range was established for the heat-transfer fluids in Table 13.

TABLE 13. HEAT TRANSFER FLUIDS

Fluid	Material	Cost, \$
Aroclors 1242	Chlorinated Biphenyl	< 5.00/gallon
Aroclors 1248	Chlorinated Biphenyl	< 5.00/gallon
Aroclors 1254	Chlorinated Biphenyl	< 5.00/gallon
Dow Corning 710	Silicone	>20.00/gallon
Dow Corning 550	Silicone	>20.00/gallon
Hitec	Inorganic Salt	< 2.00/lb
Humble Therm 500	Hydrocarbon	< 2.00/gallon
Hydrotherm 700-10A	Aryl Silicate	<20.00/gallon
Hydrotherm 700-155	Aryl Silicate	<20.00/gallon
Hydrotherm 700-160	Aryl Silicate	<20.00/gallon
Mobiltherm Light	Hydrocarbon	< 2.00/gallon
Mobiltherm 600	Hydrocarbon	< 2.00/gallon
Mobiltherm 603	Hydrocarbon	< 2.00/gallon
Mobiltherm 605	Hydrocarbon	< 2.00/gallon
Mercury	Metal	< 5.00/lb
Sun 21 Oil	Hydrocarbon	< 2.00/gallon
Thermofluid	Animal Oil	< 5.00/gallon
Tri Aryl Borate	Organic Salt	< 5.00/gallon
Ucon Fluid	Polyalkylene Glycol Derivative	< 5.00/gallon

Jet Fuel

The United States and the Soviet Union have supported a large research effort to improve the thermal stability of gas-turbine aircraft fuels. Various programs have been funded to investigate the thermal stability of jet fuel and to develop refining methods and additives to improve this property. Jet fuels would be more uniform from batch to batch and might well provide a better source of a low-cost heat-transfer agent.

The Soviet Union has announced the use of two new jet fuels. A review of the composition and physical properties of these would also be in order to evaluate these two fuels as possible heat-transfer candidates.

Special efforts have been made by fuel researchers to develop exotic fuels for advanced aircraft. A review of this work could very well uncover a material suitable for use as a heat-transfer fluid in the range of 500 - 600 F.

Hydraulic Fluid

Long after the United States switched to synthetic fluids in its aircraft hydraulic systems, the Soviet Union continued to use AMG-10, a mineral oil fluid. AMG-10 hydraulic fluid is an excellent material and a review of technical data pertaining to its chemical and physical properties to evaluate its possible use as a heat-transfer fluid is advised.

Inorganic Salts

An inorganic salt, "Glauber's salt" ($\text{Na}_2\text{SO}_4 \cdot 10\text{H}_2\text{O}$) melts at 91.4 F, is plentiful and sells for approximately \$35 per ton in carload lots. Due to the low cost of this material and its availability, a study of its thermophysical properties to evaluate its use as a heat-transfer fluid would seem advisable.

APPENDIX A

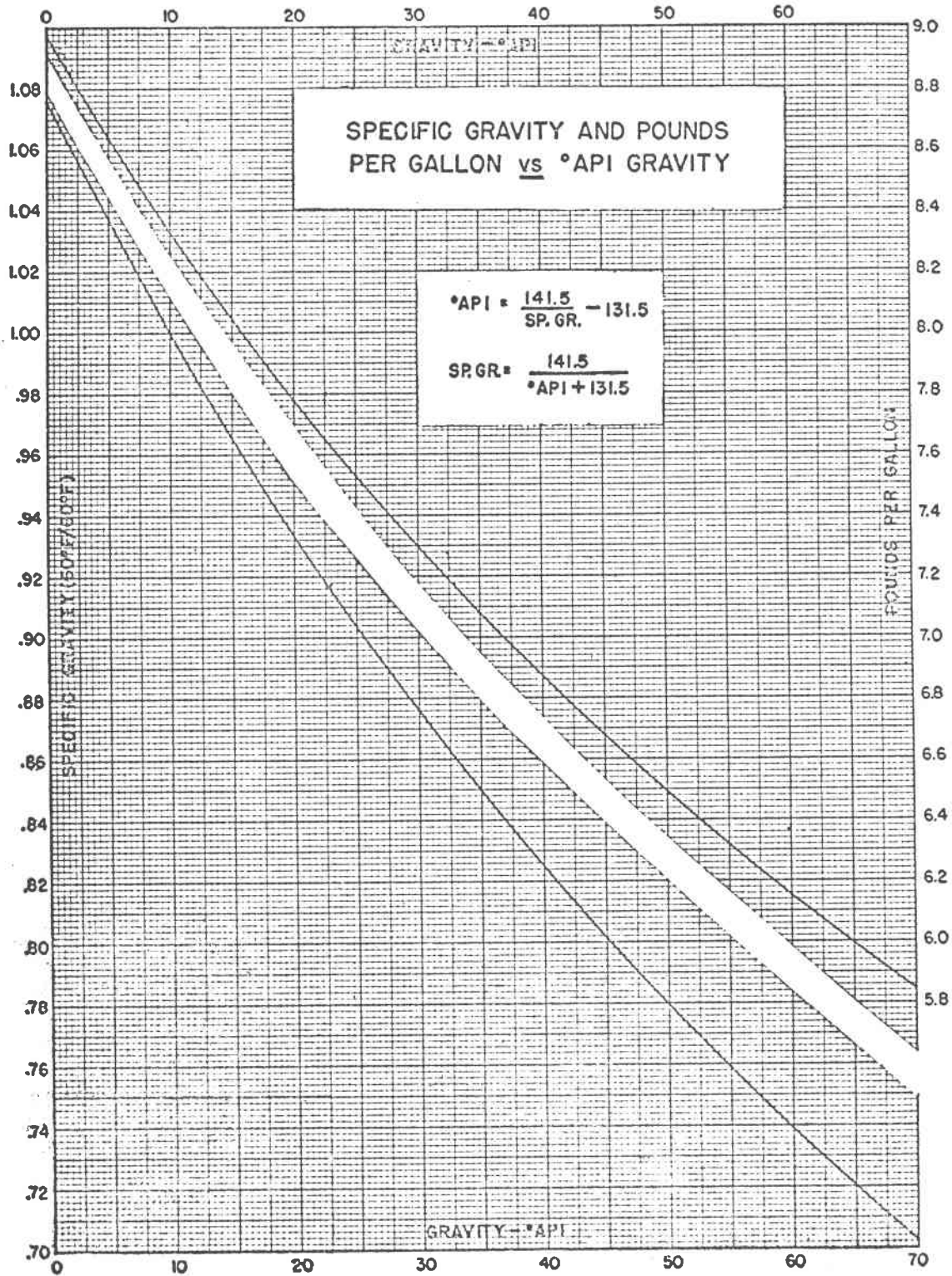
SPECIFIC GRAVITY AND POUNDS PER GALLON VS

DEGREES API GRAVITY (1)

(1) From Maxwell, J. B., "Data Handbook on Hydrocarbons", D. Van Nostrand Company, Inc., New York (1950), p 138.

APPENDIX A

SPECIFIC GRAVITY AND POUNDS PER GALLON VS
DEGREES API GRAVITY

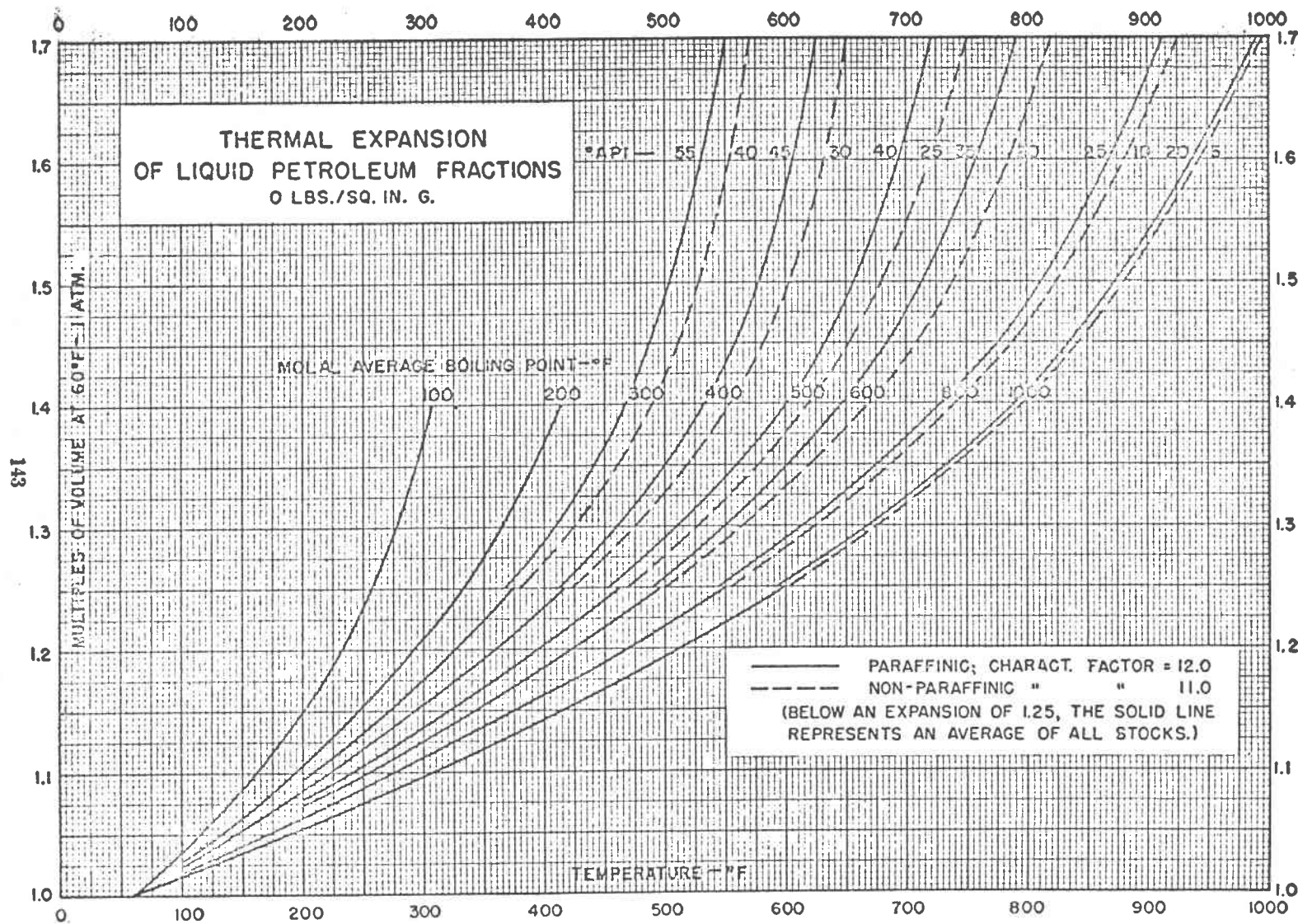


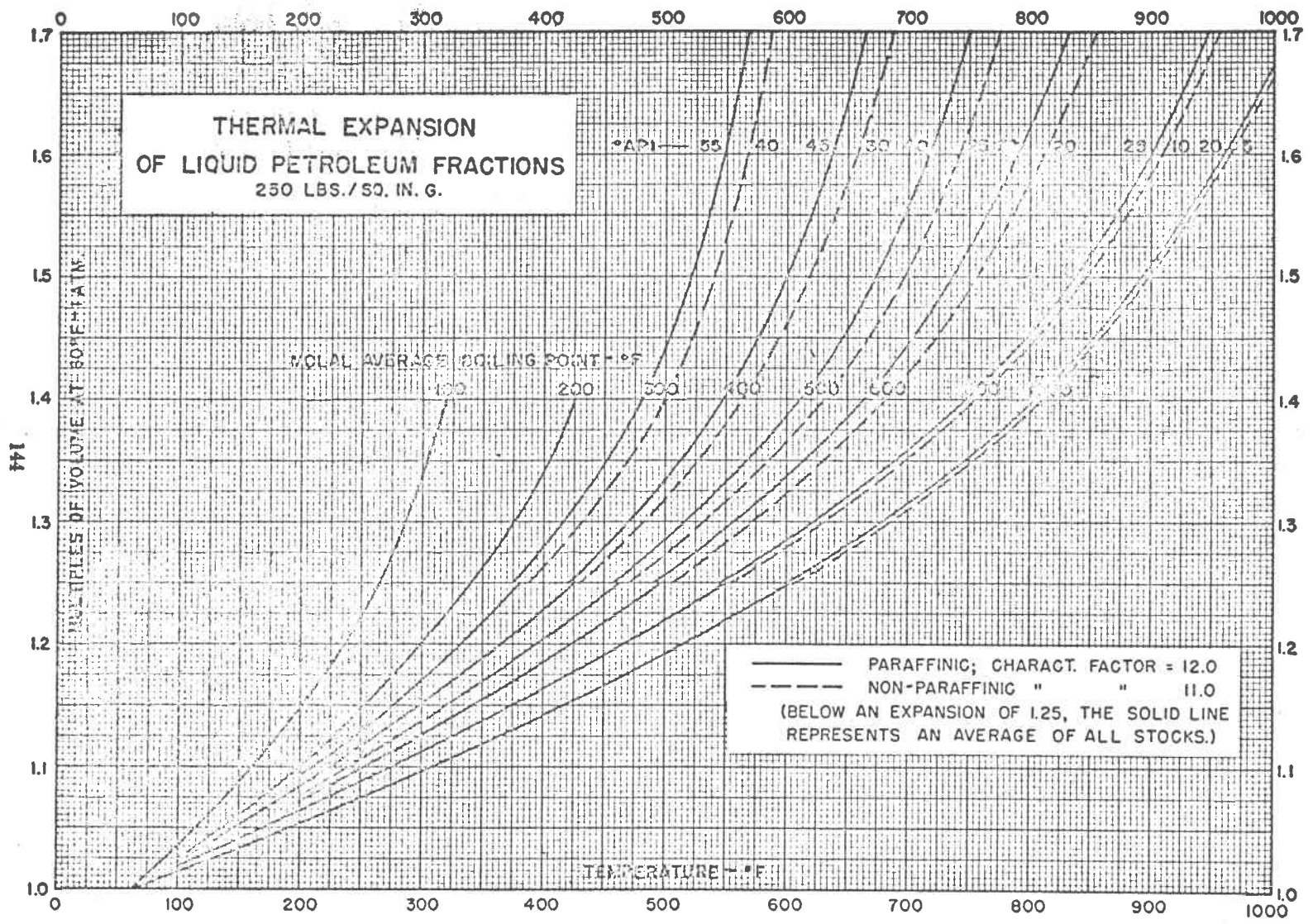
APPENDIX B

THERMAL EXPANSION OF LIQUID PETROLEUM FRACTIONS⁽¹⁾

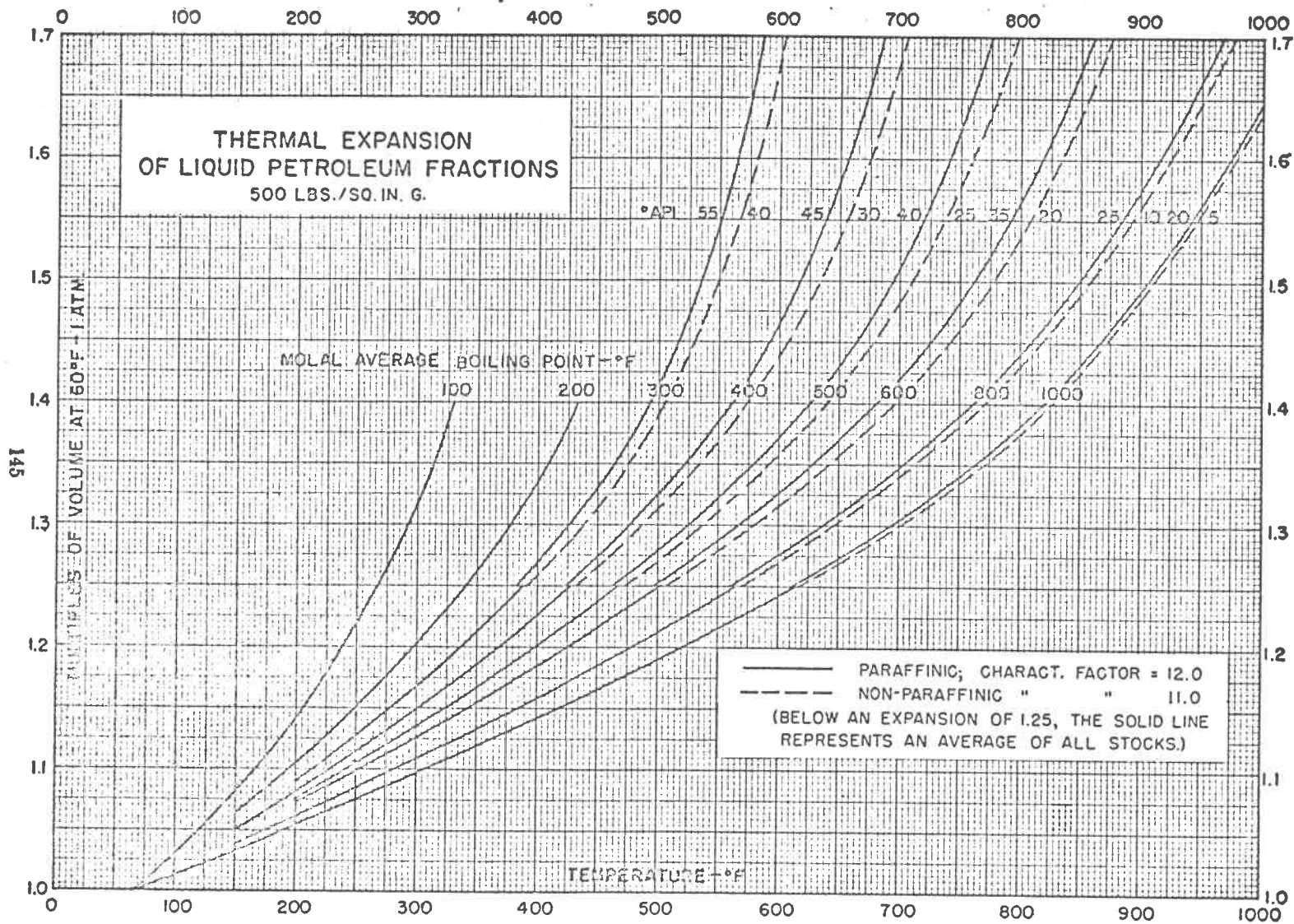
(1) From Maxwell, J. B., "Data Book on Hydrocarbons", D. Van Nostrand Company, Inc., New York (1950), pp 143-147.

THERMAL EXPANSION OF LIQUID PETROLEUM FRACTIONS

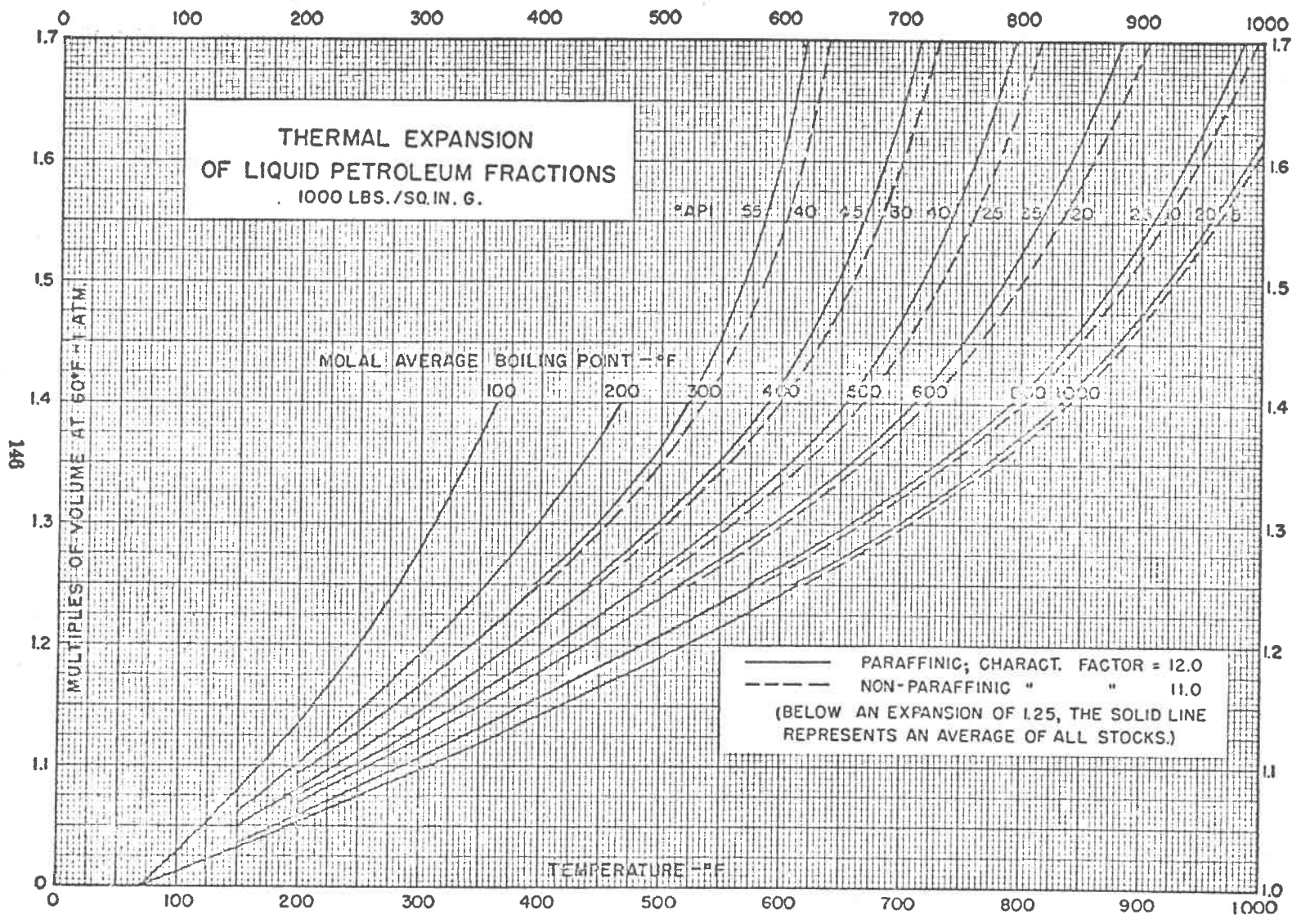




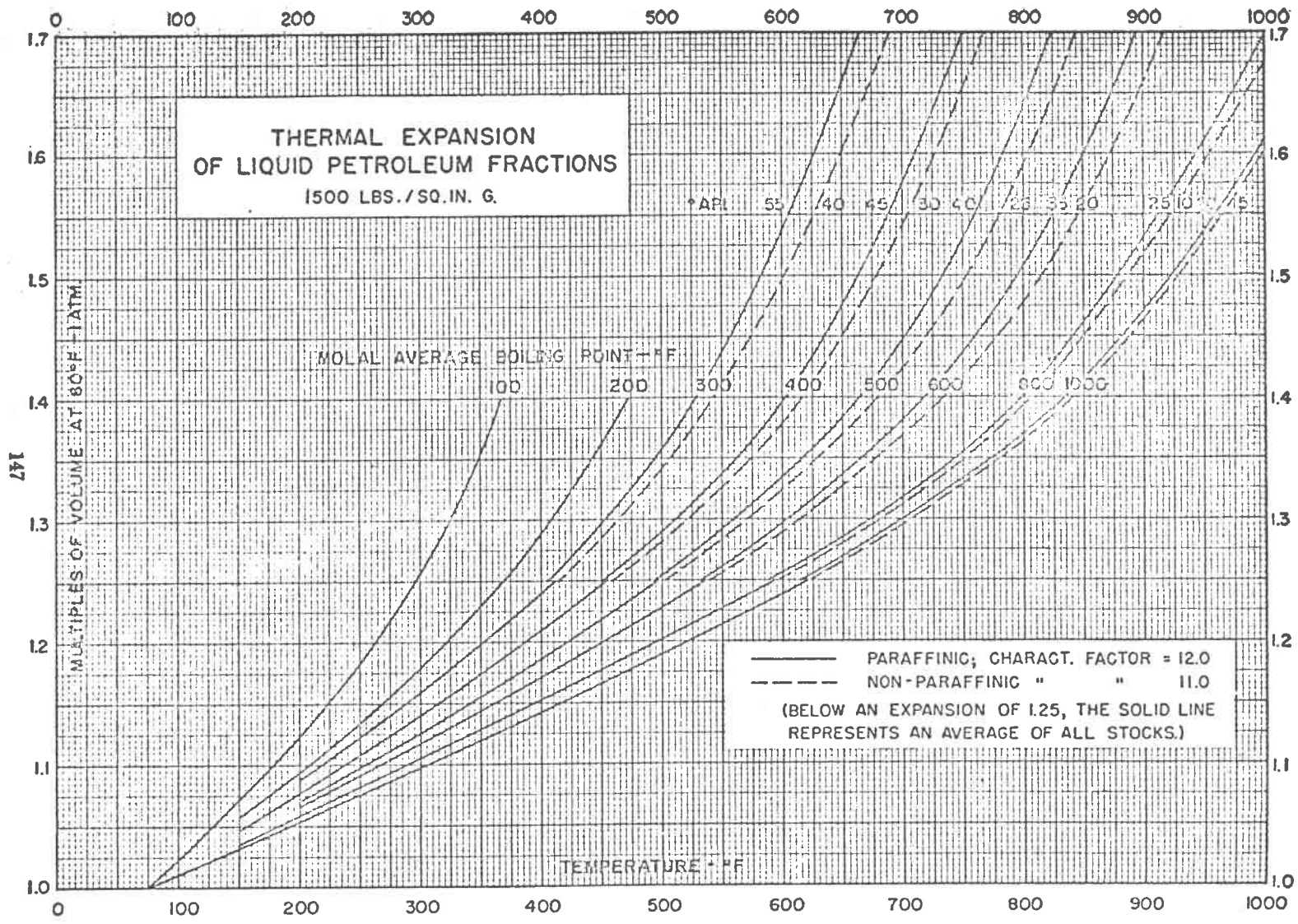
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B-4



**THERMAL EXPANSION
OF LIQUID PETROLEUM FRACTIONS**
1500 LBS./SQ. IN. G.



B-5

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APPENDIX C

BIBLIOGRAPHY OF RECENT CHEMICAL ABSTRACTS RELATED

TO FUEL OIL ADDITIVES (1)

(1) From Chemical Abstracts Service

APPENDIX C

BIBLIOGRAPHY OF RECENT CHEMICAL ABSTRACTS RELATED TO FUEL OIL ADDITIVES

127967g Fuel oil sludge-dispersant composition. Adams, Alan Douglas (Atlas Chemical Industries, Inc.) U.S. 3,751,234 (Cl. 44/66; C 10l), 07 Aug 1973, Appl. 112,065, 02 Feb 1971; 3 pp. Sludges which normally form in fuel oil are maintained in suspension by the addn. of a dispersant comprising a partially esterified polyethoxylated or propoxylated aniline-HCHO resin having low mol. wt. Thus, an ethylene oxide-aniline-HCHO polymer esterified with acetic and oleic acid was useful as dispersant when added to fuel oil at 5 g/100 ml. W. R. Averett

127966f Sludge-dispersant compositions. Adams, Alan Douglas (Atlas Chemical Industries, Inc.) U.S. 3,751,236 (Cl. 44/66; C 10l), 07 Aug 1973, Appl. 151,964, 10 Jun 1971; 5 pp. Compns. having polyoxyalkylene 1-8, fatty acid ester 1-4, and H₂PO₄ 1-2 groups per mol. are useful as ashless fuel oil sludge dispersants. A sorbitol dioleate was prepd by reacting polyoxpropylene sorbitol 273 (prepd. from propylene oxide and sorbitol in 4:1 molar ratio) with oleic acid 1988 parts by wt, reacting with P₂O₅ slurried in PhMe, and reacting 342.8 parts of the product with isopropylamine 31.2 parts. In a pumping test described, 98% flow was obtained when the product was added to fuel oil at 0.013 g/100 ml, compared to 14% flow for the untreated oil. The dispersant does not emulsify water appreciably. W. R. Averett

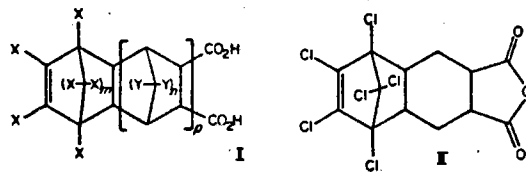
46030y Fuel oil additive. Bouvin, Jacques; Chavet, Bernard (Esso Standard Societe Anon. Francaise) Ger. Offen. 2,224,734 (Cl. C 10l), 07 Dec 1972, Fr. Appl. 71 18,538, 24 May 1971; 12 pp. 1,2-Diphenylethane (I) was used as additive for domestic fuel oils for decreasing the C deposits on the grate and in the combustion chamber. Thus, addn. of 0.2 and 0.5% I to com. fuel oil decreased the amt. of deposits by 20 ± 7 and 28 ± 4%, resp.

68424c Fuel oil blending to improve pour reduction. Cote, Edmund Wallace; Bialy, Jerzy J.; Sweeney, William M. (Texaco Development Corp.) Brit. 1,314,918 (Cl. C 10), 26 Apr 1973, Appl. 33,984/71, 20 Jul 1971; 6 pp. Addn. to Brit. 1,254,401 (see Ger. 2,028,733, CA 74:55956z). Stable blends of ethylene-vinyl fatty ester copolymers (Elvax 150, 250, 260, 300, 350, and 450 contg. 12.36-16.80 wt.% wax), a 90-100°F-pour point residual fuel oil, and a low-wax, 40°F-pour point residual fuel oil, prepd. by dispersing the copolymers in one oil at 200-350°F and adding the second oil, had pour points lower than the corresponding copolymer-free blends. Fuels blended at 350°F gave a smaller pour point depression than fuels blended at 200°F. A mixt. contg. 60:40 vol.% blend of F/18 fuel oil (API gravity 22.3, wax content 16.80 wt.%, and S 0.96 wt.%, with No. 6 fuel oil (API gravity 12.3, wax content 3.05 wt.%, and S 0.74 wt.%, and 0.125% Elvax 250 (27-9% vinyl acetate content) had pour point 53°, compared with 80° for the Elvax 250-free blend. The Elvax 250-contg. blend had a pour point that was stable 3 months.

18662n Organic compositions containing tetraoctyl (dimethylamino)methylenediphosponate as an antiwear agent. Comnichau, Axel Klaus (Mobil Oil Corp.) U.S. 3,656,036 (Cl. 252-49.9; C 10m), 03 Oct 1972, Appl. 30,573, 21 Apr 1970; 3 pp. Addn. of 0.1-5% tetraoctyl (dimethylamino)methylenediphosponate (I) improved the antiwear properties of fuel oils, lubricating oils, and greases. A product consisting mainly of I was prepd. by addn. of 0.1 mole freshly distd. di-n-octyl phosphite to 0.1 mole dimethylformamide dimethyl acetal, storing in a closed flask at room temp. for 14 hr, removing low-boiling fractions by distn. to ~100° at 14 torr and vacuum distg. the residue. In FZG test A-8.3-90, a sample of hydrogenated polydecene gear oil withstood 8 stages without an additive, but with 0.5% by wt. I, it withstood 11 stages. In a std. 4-ball wear test at 60 kg for 3 min, 390°F and 1800 rpm, a pentaerythritol base ester of 5cSt viscosity at 210°F gave a wear scar of diam. 2.24 mm. When I in an amt. equiv. to 0.04% by wt. P was added to the ester, the wear-scar diam. was only 0.8 mm.

W. R. Averett

51012c Reaction products of a polyamine and carboxylic acids for use in fuels, lubricating oils, greases, and plastics. Cyba, Henryk A. (Universal Oil Products Co.) U.S. 3,669,289 (Cl. 252-51.5A; C 10m), 02 May 1972, Appl. 37,941, 15 May 1970; 7 pp. The products are formed by condensation of a polyamine



contg. ≥ 2 primary and (or) secondary N atoms, a polyhalo-polyhydro polycyclic dicarboxylic acid of general formula I, in which X is Cl and (or) Br, H, or a C₁₋₄ alkyl group, ≥ 2 of the X's being halogen atoms, Y is a halogen atom, H, or a C₁₋₄ alkyl group, $m = 1-4$, $n = 0-4$, and $p = 0-4$, or deriv. and a halogen-free dicarboxylic acid or deriv. Thus, 0.5 mole dodecylsuccinic anhydride was mixed with 0.5 mole 5,6,7,8,9,9-hexachloro-1,2,3,4,4a,5,8,8a-octahydro-5,8-methano-2,3-naphthalenedicarboxylic anhydride (II) and dissolved in 400 g hot Ansul 141 (diethylene glycol dimethyl ether) and 400 g toluene. This mixt. was added slowly to a refluxing mixt. of 0.5 mole diethylenetriamine and 200 g toluene during 3 hr. After 5 hr refluxing, the mixt. was decolorized and the solvents removed under vacuum. The residue was recovered as 355 g resinous powder sol. in acetone and contg. 4.85% N and 25.6% Cl. It was useful as a detergent-dispersant in fuel oil. Similar products were used in flameproofing of an acrylonitrile-butadiene-styrene copolymer and an EPDM rubber, as lubricant additives, and as an insecticide.

Walter G. Toekelt

18602i **Stabilization of organic substances with a cyclic borate of a polymeric alkanolamine.** Cyba, Henryk A. U.S. 3,692,680 (Cl. 252-49.6; C 10m), 19 Sep 1972, Appl. 780,601, 02 Dec 1968; 4 pp. Continuation-in-part of U.S. 3,598,855 (CA 75:152555w). Cyclic borates of polyalkanolamines are useful as stabilizers for hydrocarbon oils, e.g. gasoline, naphtha, kerosine, gas oil, fuel oil, or lubricating oil, and for plastics, resins, and rubbers. Thus, 2 moles hydrogenated tallow amine (Alamine H26D) was treated with 1 mole epichlorohydrin in iso-PrOH and NaOH. The product was a white amorphous solid having a softening point of 45° and m. ~67° to give a clear, light-yellow liq. Cyclic borates was prepd by refluxing 1 OH equiv. of the polymeric alkanolamine with 0.5 mole Me₃B. Fuel oil contg. 0.001% by wt. of the borate showed no rise in the differential pressure across a filter after 300 min. in the ASTM-CRF Fuel Coker Test, compared with a differential pressure of 25 in Hg in <100 min without the additive. In another example, 0.5% by wt. of a cyclic borate in a butadiene-styrene synthetic rubber reduced the deterioration due to oxidn. and (or) O.
F. M. Mason

86846a **Possible lowering of the pour point of prospective types of liquid fuel.** Egle, Z. (Latv. S.-K. Akad., Jelgava, USSR). *Latv. Lauksaimn. Akad. Raksti* 1971, No. 35, 179-84 (Latvian). Additives B-701 and B-704, developed by the All-Union Research Institute of the Oil Industry, gave good results when used as pour-point depressants for addn. to exptl. furnace fuels (thermocracking and coking distillates). B-701 in a concn. of 0.5% lowered the pour point of a fuel sample 8°; 1% of its additive lowered it 18°. During prolonged storage of fuels contg. the pour-point depressant, stratification may take place; therefore, the fuel oils should be mixed or pumped once a month.
P. Alksnis

86971n **Middle distillate fuel having improved low-temperature flowability.** Feldman, Nicholas (Esso Research and Engineering Co.) Brit. 1,301,933 (Cl. C 10l), 04 Jan 1973, US Appl. 807,966, 17 Mar 1969; 6 pp. Addn. to Brit. 1,301,326. Low-temp. flow properties of middle distillate fuel oil are improved by the addn. of 0.01-0.1% C₂H₄ copolymer pour-point depressant and 0.2-1% satd. hydrocarbon (mol. wt. 600-3000 and contg. no normal paraffins). The copolymer is prepd. by adding C₂H₄ to a reactor which contg. a solvent, and unsatd. ester, such as vinyl acetate, and a peroxide free-radical catalyst. The satd. hydrocarbon can be prepd. by deasphalting a petroleum residue, adding C₃H₈, and cooling and filtering the mixt. When a middle-distillate fuel oil of -50F pour point was treated with 0.01-0.02% C₂H₄-vinyl acetate copolymer and 0.1-0.4% hydrocarbon additive, 100% recovery was obtained on filtering through a 40-mesh screen, and pour point was reduced to -20 to -40°F.
W. R. Averett

100215c Antistatic additives for hydrocarbon fuels. Filachek, Lawrence A.; Rakow, Marvin S. (Cities Service Oil Co.) U.S. 3,674,450 (Cl. 44-55; C 10/), 04 Jul 1972, Appl. 136,993, 23 Apr 1971; 6 pp. The antistatic additives consist of a synergistic mixt. of (a) a component prepd. from 3 parts by wt. of the condensation product of 1 mole dimer acid and 2 moles *N*-tallow-1,3-propylenediamine with 1 part of a mixt. of 65% diisooctyl hydrogen orthophosphate and 35% isooctyl dihydrogen orthophosphate and (b) the mixed Ba and Zn salts of a mixt. of C₁₆₋₂₄ naphthenic acids and 2-ethylhexanoic acid, together with the monobutyl ether of ethylene glycol. The ratio of component b to component a is 1-15:1. The hydrocarbon fuel should be a distillate fuel, such as no. 1 or no. 2 fuel oil, diesel fuel, or turbine fuel and should contain 3-25 parts additive per 1000 bbl. The fuel oil compns. have thermal and oxidative stabilities and good H₂O tolerance. Correction of CA 75:128711q.

116589t Middle distillate of improved pour point. Gaydasch, Alexander (Universal Oil Products Co.) U.S. 3,681,038 (Cl. 44-66; C 10/), 01 Aug 1972, Appl. 17,860, 09 Mar 1970; 3 pp. The pour points of middle distillates were improved by the addn. of an *N,N*-dialkyricinoleamide in which ≥ 1 alkyl group contained 12-26 C atoms. A typical additive is *N,N*-ditallow ricinoleamide (I), prepd. by refluxing ricinoleic acid and *N,N*-ditallow amine in diethylbenzene contg. Amberlist 15 resin. Other forms of the additive were prepd. by replacing the tallow amine with Kenamine S-190 (a mixt. in which the alkyl groups are arachidic and behenic), or the product of ditallow amine and castor oil. Pour-point depressions ranged from 40 to 50°F when I was added at 1000 ppm to middle distillates with initial pour points of 5-10°F.
W. R. Averett

7688v 2-(Cyclohexylamino)-2-penten-4-one gasoline stabilizer. Hironaka, Yoshio; Asai, Nobumichi Japan. 71 37,178 (Cl. C 10/), 01 Nov 1971, Appl. 70 41,616, 18 May 1970; 1 pp. The title compd. (I) is effective in preventing oxidative reactions, such as the formation of gummy material on storage or transportation of gasoline, in concn. of 0.0001-1%, preferably 0.001-0.01%. The compd., easily prepd. by condensation of acetylacetone with cyclohexylamine, can be safely used with other gasoline additives. Thus, the addn. of 0.002% I decreased the formation of insol. material from a cracking gasoline by ~90%.
T. Higa

6333t Middle distillate with improved filterability and flow behavior. Hnyckyj, Stephan; Cole, Charles O. (Esso Research and Engineering Co.) Ger. Offen. 2,206,719 (Cl. C 10/), 05 Oct 1972, US Appl. 115,714, 16 Feb 1971; 32 pp. 0.001-0.5% Of a mixt. contg. 1-20 parts of a wax crystn. improving polymer and 1-99 parts of a wax crystn. inhibiting polymer is added to a fuel mixt... a highly paraffinic middle distillate. The copolymers are ethylene-vinyl acetates, having different mol. wts. and they were used to improve filterability of diesel fuels and fuel oils.
C. H. Brieger

37355k Additives for increasing the low-temperature flowability of middle distillate fuel. Hnyckyj, Stephan (Esso Research and Engineering Co.) U.S. 3,660,057 (Cl. 44/62; C 10/), 02 May 1972, Appl. 807,952, 17 Mar 1969; 5 pp. The low-temp. fluidity of a waxy-cloudy middle-distillate petroleum fuel, e.g. fuel oil, through flow lines and filters is improved by addn. of 0.001-1.0 wt. % of a flow improver, e.g. a copolymer of C_2H_4 with an unsatd. monomer, and 0.01-0.099 wt. % satd. hydrocarbons (av. mol. wt. 600-30,000). Thus, the passage of a wax-cloudy fuel through screens as measured by a filterability test was improved when a 2:1 blend by wt. of flow improver (copolymer of 65% C_2H_4 and 35% vinyl ester) and a solid hydrocarbon was incorporated in the fuel.
Henry H. Ginsberg

44178z Oil-soluble, rust-preventive composition. Izumi, Kaichi; Watanabe, Takao (Kao Soap Co., Ltd.) U.S. 3,720,615 (Cl. 252/33; C 10m), 13 Mar 1973, Japan. Appl. 69 63,464, 11 Aug 1969; 5 pp. A binary rust-preventive compn. is prepd. for use in hydrocarbon oils, such a fuel oils or lubricating oils. The compn. consists of (a) a C_{10-14} poly(carboxylic acid) contg. 2 or 3 carboxyl groups or a partial ester of the acid with a C_{10-18} alc. and (b) an aliph. tertiary amine having 3 hydrocarbon groups, each of which has $\geq 1-20$ carbon atoms and ≥ 1 of which has 6-20 carbon atoms. The wt. ratio of a and b is from 95:5 to 5:95. An alk. earth metal alkylbenzenesulfonate is added as a dispersant. The preferred acids are diisobutylene- and tetraisopropylene-succinic acid, and the dispersant is Ca alkylbenzenesulfonate. The compn. also gives improved demulsibility to the hydrocarbon oils.
B. L. McCully

161881p Fuel oil compositions. Kingsland, Michael; Price, Roger Charles (Esso Research and Engineering Co.) *Brit. 1,308,046* (Cl. C 10), 28 Feb 1973, Appl. 41,557/69, 20 Aug 1969; 5 pp. Pour-point depressants for oils with a viscosity of 15-2000 cSt at 100°F were prepd. by reacting maleic anhydride with an unsatd. polymer and esterifying the product with behenyl alc. Thus, 0.1 wt. % of the bibehenyl ester of the reaction product lowered the pour point of a fuel oil from 45 to 25°F.

86964n Fuel oil composition. Kober, Alfred E.; Rossi, Albert (Esso Research and Engineering Co.) *Fr. 2,114,718* (Cl. C 10), 04 Aug 1972, US Appl. 90,115, 16 Nov 1970; 25 pp. The f.p. of fuel oil b. 121-399° is decreased by addn. of 0.001-0.5% of an C₂H₄-vinyl ester copolymer and 0.001-0.5% of a sorbitan ester, such as the stearate. The results of extensive exptl. work with 5 different fuel oils and various additives show the extent of f.p. lowering. Thus, a fuel oil having a 10% b.p. 195°, 90% b.p. 339°, amline point 73.0°, a pour point of -15°, and a cloud point of -3.3° mixed with a 5:1 molar C₂H₄-vinyl acetate copolymer and sorbitan monopalmitate had a cloud point of -14.4°.

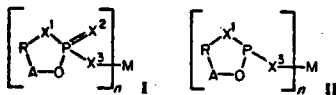
L. F. Dixon

50990h Material use and corrosion protection in the production and utilization of fuel oils. Lange, M. (Leipzig, Ger.). *Freiberg. Forschungsh. A* 1972, 503, 35-50 (Ger). Problems arising in manufg. fuel oils from S- and V-contg. petroleum and in using the oils in industrial furnaces are discussed. Corrosion of metal parts of the distn. unit in distg. the petroleum at >250° is prevented by using Cr-steel distn. columns. Duroplastic layers are expected to afford adequate protection against corrosion by HCl. When burning V-contg. heavy fuel oils furnace parts of a 50/50 Cr-Ni alloy are corrosion resistant even at >600°.

J. S. Buii

101882q Hydrocarbon fuel or oil compositions containing polyesters as pour point depressants. Latham, Kenneth Geoffrey; Price, Roger Charles (Esso Research and Engineering Co.) *Brit. 1,308,626* (Cl. C 10), 28 Feb 1973, Appl. 25,990/70, 29 May 1970; 8 pp. Polyester additives, prepd. by reacting alkene succinic anhydrides, pentaerythritol, behenyl alc., and F acid (C₂₀₋₂₂ carboxylic acid) were used to lower the pour point of residual fuels with viscosity > 15 cSt at 100°F. Thus, 0.3 wt. % of this polyester depressed the pour point for a north Africa waxy residuum having a viscosity of ~350 cSt at 100°F from 105 to 36°F.

6317r Cyclic phosphorus ester salts as antioxidant detergents for lubricating oils and fuel oils. Morris, Peter John; Simpkin, Dennis John (Esso Research and Engineering Co.) Brit. 1,282,999 (Cl. C 07f, C 10lm), 26 Jul 1972, Appl. 56,511/70, 27 Nov 1970; 5 pp. Detergent oxidation inhibitors for



lubricating oils are prepd. having the formulas I or II, in which X¹, X², and X³ are the same or different and are O or S atoms; R is a C- and H-contg. aryl group; A is a C- and H contg. group, M is a metal, an NH₄ group, or a salt-forming amine group; and n is the valency of M. For example 130 g dodecylphenol and 40 ml HCHO soln. were added to 20 g of an aq. 50% NaOH soln. and the mixt. was held at 40° for 6 hr. HOAc was added to neutralize the alkali and the org. layer was washed with water before vacuum stripping to give dodecylsaligenin. P₂S₅ (9.7 g) and 25 g dodecylsaligenin were mixed in xylene and heated at 100° for 1 hr. Ca(OH)₂ (10 g) was added with 1 ml H₂O as catalyst. The H₂O from the reaction and that used as catalyst were removed by azeotropic distillation. Solvent removal in filtration gave a 50 wt. % concentrate in mineral oil. In an oxidation-inhibition test, a lubricating oil compn. contg. more wt. % of the inhibitor than one not contg. an inhibitor were held at 340°F for 23 hr with air passing through the oil at 2 ft³/hr. The viscosity of the oil contg. the inhibitor increased 10% while that of the oil not contg. an inhibitor increased 214%.

O. C. Braids

81434a Surfactant additives for heavy oil and other petroleum products. Nagata, Akira (Tokai Oil Manufg. Industry Co., Ltd.) Japan. Kokai 73 42,004 (Cl. 18 E21, 18 E0, 18 F0), 19 Jun 1973, Appl. 71 73,369, 29 Sep 1971; 3 pp. Amphoteric surfactants [RCONH(CH₂CH₂NH)_{n-10}CH₂CH₂NR'₂CH₂CO₂]_nM (R = n-C₂₋₃₀ satd. and unsatd. fatty acid residue, R' = H or R, n = 2-4, M = H, Mg, Cu, Si, Zn, Ca, Al, or Ba) were useful as additives for heavy oil and other petroleum products and promoted sludge dispersion, combustion, SO₂ abatement, water sepn., and corrosion resistance. Thus, 282 g oleic acid and 146 g triethylenetetramine were heated at 170° for 5 hr, treated with 117 g ClCH₂CO₂Na (as 40% soln.) at 40° for 4 hr, heated with 95 g MgCl₂ (as 10% aq. soln.) for 2 hr, and concd. in vacuo to give additive (I). In combustion of bunker C heavy oil (150 kg/hr, air ratio 1.3), the SO₂ formation was reduced from 1300 to 600 ppm and soot accumulation (after 20 days) from 0.016 to 0.00183 g/cm² by the presence of 0.2% I.

32420p Stabilizers for distillate hydrocarbon fuel oils. Rakow, Marvin S.; Konig, Robert J. (Cities Service Oil Co.) U.S. 3,701,641 (Cl. 44-73; C 10l), 31 Oct 1972, Appl. 854,266, 29 Aug 1969; 4 pp. Distillate fuels, such as diesel and jet fuels, are stabilized by an additive contg. a polyamine with ~2-6 amino groups and 24-50 C atoms, N,N'-disalicylidene-1,2-propylenediamine (I), and a cyclohexylamine. Thus, a rather unstable No. 2 fuel oil was mixed with various additives (expressed as PTB: lb/1000 bbls), and was oxidized 192 hr at 180°F, where the color (ASTM D-1500) was considered a measure of stability. Thus, 3.0 PTB of dimer diamine (II) derived from a dimerized C₁₈ monocarboxylic acid used alone, the insols. were 7.03 mg/100 ml and the color was 5.5. With I 3.0, II 3.0, and N,N'-dimethylcyclohexylamine 3.0 PTB, the insols. were 1.12 mg/100 ml, and the color was 3.0.

W. R. Averett

113609j Role of fuel oil additives as a sludge preventing factor. Rauhala, Veikko T. (Finnish Oil Res. Lab., Tech. Res. Cent. Finland, Helsinki, Finland). *Tech. Res. Cent. Finl., Mater. Process Technol., Publ.* 1972, No. 6, 28 pp. (Eng.). Five fuel-oil additives, randomly selected, were evaluated at 1:100 and 1:2000 concns. in a 1:1 blend of light and heavy fuel oils. None of the additives affected the surface tension of the oil. Sludge formed in additive-free oil in the presence of O₂ but not in the presence of N₂. The soln. of O₂ in the oil appeared to be very rapid. The additives were most effective in retarding sludge formation by O₂ at 2 and 4-day exposure. Under normal conditions (alternating between 24° and 100° for 16 weeks, with an air atm.) ir examn. showed increasing absorbance at 1705 cm⁻¹ (representing aliph. acids, ketones, aldehydes, and esters), but the absorbance was not proportional to the amt. of sludge formed. The sludge formed under normal conditions was more gelatinous than the material formed in an O₂ atm. The additives act as reaction inhibitors or sludge preventers without aging. W. R. Averett

86824s Depolymerization resistance of the viscous additive, copolymer of isobutylene with isopropenyl-p-xylene. Sadyk= hov, Z. A.; Khanlarov, T. G. (Azerb. Gos. Univ. im. Kirova, Baku, USSR). *Khim. Tekhnol. Topl. Masel* 1972, 17(10), 20-3 (Russ). The depolymn. resistance decreased with the mol. wt. increase of the copolymer. The introduction of an arom. ring into the polyisobutylene increased its thermal depolymn. resistance. Soln. of the title copolymer in turbine oil was used for the expts. The copolymer having 95:1 mole ratio and ~5000 mol. wt. was the best additive for improving the viscosity-temp. properties of oils.

86824s Depolymerization resistance of the viscous additive, copolymer of isobutylene with isopropenyl-p-xylene. Sadyk= hov, Z. A.; Khanlarov, T. G. (Azerb. Gos. Univ. im. Kirova, Baku, USSR). *Khim. Tekhnol. Topl. Masel* 1972, 17(10), 20-3 (Russ). The depolymn. resistance decreased with the mol. wt. increase of the copolymer. The introduction of an arom. ring into the polyisobutylene increased its thermal depolymn. resistance. Soln. of the title copolymer in turbine oil was used for the expts. The copolymer having 95:1 mole ratio and ~5000 mol. wt. was the best additive for improving the viscosity-temp. properties of oils.

126478r Burner fuel additives. Salooja, K. U. (Esso Res. Cent., Abington/Berkshire, Engl.). *Combustion* 1973, 44(7), 21-7 (Eng). Burner-fuel additives used to control smoke emission, high-temp. corrosion by metal impurities in the fuel, and low-temp. corrosion by metal impurities in the fuel, and low-temp. corrosion caused by acid sludge from S in the fuels is reviewed with 41 refs. J. F. Schultz

7633y New polymeric pour-point depression additives. Samoilov, S. M.; Monastyrskii, V. N. (Vses. Nauchno-Issled. Inst. Pererab. Nefti, USSR). *Khim. Tekhnol. Topl. Masei* 1973, 18(2), 60-2. (Russ). A review with 23 refs.

74539q Oil compositions. Shell Internationale Research Maatschappij N. V. *Neth. Appl.* 72 02,748 (Cl. C 10f, E 21b), 07 Sep 1972. *Brit. Appl.* 6132/71, 05 Mar 1971; 13 pp. Polymers contg. heterocyclic groups and C₁₆₋₂₆ alkyl side chains are effective pour-point depressants for waxy crudes. Particularly suitable are the copolymers of 4-vinylpyridine and alkyl acrylates. Thus, a mixt. of eicosyl and docosyl acrylate was polymd. under programmed addn. of 4-vinylpyridine, to yield a polymer with av. mol. wt. of 40,000. The alkyl acrylate - 4-vinylpyridine ratio was 1:0.3. Addn. of 400 ppm of this copolymer to a waxy crude decreased the pour point from 24 to 12°, when measured under a cooling rate of 30°/hr. With a 5°/hr cooling rate, the pour point was lowered to 0°. R. J. Corbeels

37296s Development and testing of additives. Scholze, S. (Freiberg, Ger.). *Freiberg. Forschungsh. A* 1972, 503, 69-79 (Ger). Mixts. of metal salts of alkylarenesulfonic acids and aromatic organometallic compds., preferably Cu compds., are added to fuel oils to inhibit sludge formation and improve their combustion characteristics. The sulfonic acids are preferably obtained by extg. acid resins (by-products of petroleum refining) with high-b.p. pyridine bases, the latter imparting to the additives corrosion-inhibiting properties. J. S. Buij

126487t Causes and prevention of corrosion of petroleum tank bottoms. Shigeno, Hayata; Okamoto, Katsutomo (Nakagawa Boshoku Kogyo Co., Ltd., Tokyo, Japan). *Sekiyu To Sekiyu Kagaku* 1972, 16(11), 46-55; 1973, (1), 112-21 (Japan). A review is given of causes, detection methods, accident cases, and prevention methods of corrosion of petroleum tank bottoms, 5 refs. T. Higa

18678x Anticorrosive additives for fuel oil. Shiraishi, Tsunenao; Edagawa, Setsuji (Shiraishi Industry Co., Ltd.) Japan 71 32,585 (Cl. C 10/ B 01/), 22 Sep 1971, Appl. 66 18,070, 22-Mar 1966; 4 pp. Powdered additives with good dispersibility are prepd. from aq. suspensions of $Mg(OH)_2$ or $Ca(OH)_2$ by using Na naphthenate, Na stearate, and/or *N*-stearylbetaine as a surfactant. Thus, an aq. suspension (of 14% solids) of $Mg(OH)_2$ (av. particle size 0.05μ) was stirred 30 min at 95° with a 5% aq. soln. contg. 5 wt. % Na stearate based on $Mg(OH)_2$, dried at $70-80^\circ$, and milled to give an additive of av. particle size 0.05μ . H. Kuroe

6329w Dispersed preparation for reducing hydrodynamic flow resistance. Stratta, Julius John; Frank, Carl William; Barrere, John Andrew, Jr. (Union Carbide Corp.) Ger. Offen. 2,208,519 (Cl. C 10m), 21 Sep 1972, US Appl. 118,442, 24 Feb 1971; 56 pp. Hydrocarbon oil contg. finely divided, H_2O -sol. ethylene oxide polymer (I); an inert, normally fluid, H_2O miscible, org. carrier, e.g. propylene glycol (II) and a suspended material, e.g. silicic acid (III), inert which retards the creep of the compn. The I has a mol. wt. $>500,000$ and particle size of <20 mesh (85 wt. %) and <60 mesh (10 wt. %). The fluid has a pour point $<-30^\circ$, flash point $>93^\circ$ and mol. wt. <160 . Thus, a mixt. of I (mol. wt. 2,500,000) 25, II 73 and III 2 parts had a viscosity of 11,650 cP and a gel strength of 13 dyne/cm². A mixt. of I 25, II 70 and III 5 had values of 50,000 and 320 and another mixt. of I 25, II 69 and III 6 had values of 156,500 and 261, resp. C. J. Boner

74488x Hydrofining of a mixture of distillates from the straight-run distillation and thermocontact cracking of sulfur petroleums to produce low-sulfur boiler fuel. Teregulov, D. Kh.; Karzhev, V. I.; Rabinovich, B. Ya.; Korshunova, L. N.; Kulikova, G. P. (Vses. Nauchno-Issled. Inst. Neft. Prom., Moscow, USSR). *Khim. Tekhnol. Topl. Mase* 1972, 17(12), 14-15 (Russ). A mixt. of the vacuum distillate of straight-run Rashkensk petroleum (fraction $350-500^\circ$) and 25% of a $350-500^\circ$ fraction of a petroleum asphalt of high-S petroleum, was purified by treatment with H at 5 atm. and 400° , using an $AlCoMo$ catalyst. The low-boiling fraction of the raffinate ($<180^\circ$) after addnl. purifn. was used as feed component for catalytic cracking. The fraction $180-350^\circ$ was unsuitable for diesel fuel, owing to high unsatn. but it was a good pour-point depressant for the fuel oil made from the fraction b. $>350^\circ$. The yield of boiler fuel (contg. 0.3% S) was 94% based on the feed. The activity of the catalyst was not impaired after 1300 hr continuous use. A. S. Leveslev

94507b Flow-improving additives for residual fuel oils. Texaco Development Corp. Fr. Addn. 2,149,588 (Cl. C 10d), 04 May 1973, Appl. 70 21,635, 12 Jun 1970; 10 pp. Addn. to Fr. 2,051,148 (See Ger. 2,028,733, CA 74:55956z). Ethylene-vinyl acetate polymers were added to mixts. of high and low pour-point residual fuel oils. Thus, the pour point of a mixt. of 60 vol. % F/18 fuel oil (22.3° API, 37.8° pour point, 16.80% wax, and 0.96% S) and 40 vol. % No. 6 fuel oil (12.3° API, 4.4° pour point, 3.05% wax, and 0.74% S) was reduced from 26.7° to 11.7° by addn. of 0.125% Elvax 250. F. M. Mason

37297c Development and effect of fuel inhibitors. Van den Heuvel, H. M. J. (Rotterdam, Neth.). *Freiberg. Forschungsh. A* 1972, 503, 51-67 (Ger). A review is given of obstacles encountered in storage and combustion of fuel oils, such as sludge deposition on tank bottoms, formation of ash layers on heat-exchanging surfaces, and corrosion of the inside of heat-transmission parts and exhausts by H_2SO_4 . These obstacles can often be disposed of by mixing the oils with "additives," i.e. chems. capable of breaking H_2O -in-oil emulsions, antioxidants, combustion catalysts, and inhibitors that decrease the oxidn. of SO_2 to SO_3 . No refs. J. S. Buij

161879u Stabilization of fuel additives. Wiley, David R. (Betz Laboratories, Inc.) U.S. 3,713,792 (Cl. 44-4; C 10d), 30 Jan 1973, Appl. 101,152, 23 Dec 1970; 4 pp. A method of stabilizing fuel additives with a glycerol ester is described. Thus, Rocket Oil B (naphthenic oil) 55.0, Alkaterge C (alkyl-substituted oxazoline cationic surfactant) 2.0, glyceryl tris(12-hydroxystearate) 0.3, Al_2O_3 22.7, MgO_2 20.0% was fed to fuel oil at 0.8 quart/1000 gal. oil. The fuel fed to a furnace remained stable and did not form any sludge. H. H. Ginsberg

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